

LANSCCE DIVISION TECHNOLOGY REVIEW

ASTERIX—A New Spectrometer for Studies of Nanomagnetism and Magnetic Properties of Complex Materials

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The development of neutron spin and sample excitation correlation techniques is essential for understanding a wide range of magnetic and electron transport problems in the emerging class of complex materials, including, for example, nanostructure-engineered and -adaptive materials. A detailed understanding of complex materials requires studies that use neutron beams to characterize the exotic magnetic and atomic structures of these materials under extreme conditions of high-magnetic fields, high pressures, and very low temperatures. Neutrons offer the unique possibility of scattering from spin and orbital moments in the material in addition to the normal scattering from atomic nuclei.

Complex instrumentation issues involved in extracting a polarized neutron beam from a pulsed neutron source and in propagating the beam through magnetic field gradients that depend upon space and time must first be understood. A solution to the polarization problems, e.g., beam splitting (the Stern-Gerlach effect), associated with interactions between (polarized) neutron beams and the equipment (in this case the high-field magnets) used to produce a variety of extreme environments simultaneously must also be developed. The solution to this problem will enable unique studies of complex materials.

Enabling the Pursuit of Scientific Frontiers in Magnetism

Traditionally, small free-standing polarizing supermirrors are used to polarize neutron beams at continuous and pulsed neutron sources. Unfortunately this approach throws away long-wavelength neutrons in favor of polarizing the short-wavelength neutrons. Why this is so can be appreciated as follows. The divergence of the neutron beam incident on the polarizing mirror is constrained (via mechanical slits) so that the neutrons reflected (or transmitted if a transmission supermirror is used) are polarized to > 95% for a particular wavelength. At a continuous source such as a reactor, the wavelength is typically $\sim 4 \text{ \AA}$ with a wavelength spread of $\sim 2\%$. At a pulsed source, that particular wavelength corresponds to the shortest wavelength of interest;

however, it is also desired to polarize neutrons with wavelengths an order of magnitude longer than the shortest within the same period of the neutron pulse. Although the cold portion of the neutron beam is also polarized by the mirror, the constraining slits defining the incident beam divergence—which are required to polarize the shortest desired wavelength neutron—block many long-wavelength neutrons that would be polarized otherwise.

The goal of ASTERIX is to produce a highly polarized intense beam of cold neutrons that has a very large cross section (2 cm by 12 cm) and covers a wide wavelength range from the Lujan Center spallation target—and to do so while minimizing the fraction of the neutron beam that is not used. The project achieves its goal by embedding polarizing transmission supermirrors inside a nickel-coated neutron guide. The neutron guide replaces the constraint of mechanical slits, which produce a neutron beam of fixed divergence, with one that constrains the beam divergence to vary proportionally with wavelength. Properly arranged within the guide, the supermirrors produce a polarized neutron beam with divergence that scales with wavelength; thus, no neutrons are wasted. Crucial to the success of this project was the acquisition of high-quality polarizing transmission supermirrors.

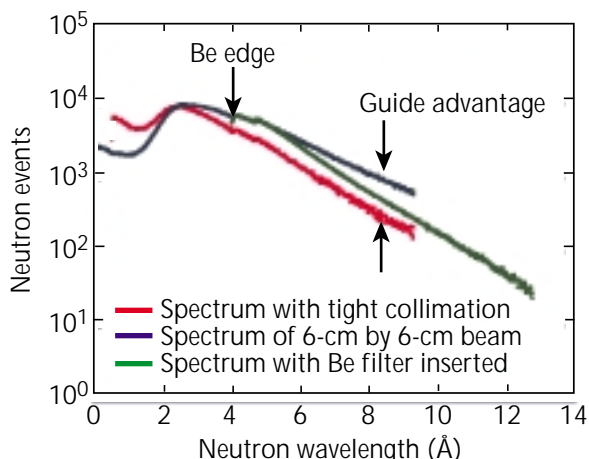
ASTERIX will allow researchers to pursue investigations of lateral and depth non-uniformity in magnetic (or electronic) heterostructures of induced (or coerced) magnetization at or near interfaces and magnetic-structure characterization. In addition to studies of nanostructured materials, ASTERIX enables research into magnetically ordered novel bulk materials—for example, diffraction measurements of materials in very high magnetic fields using cold neutrons.

Investigating the Influence of Measurement Conditions

ASTERIX received first beam during the last 48 hours of the 2000 run cycle. This beam allowed us to determine that a cooled block of beryllium does a

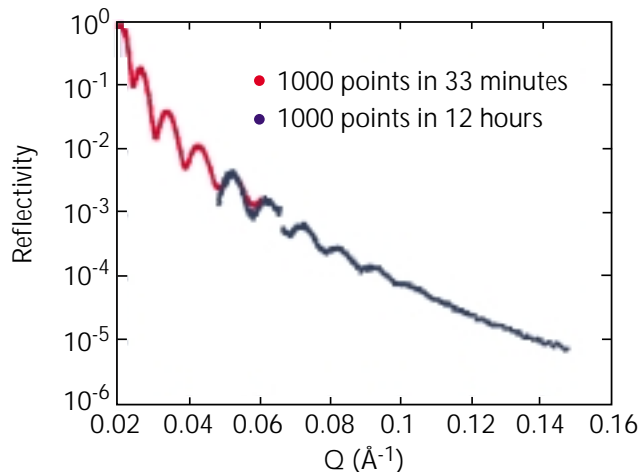
very good job removing undesirable high-energy neutrons and that our slits are opaque to neutrons. In addition, we demonstrated extraction of a neutron beam with modest polarization.

During the first week of the 2001 run-cycle, the red spectrum (red curve, Fig. 1) was measured for a highly collimated beam (achieved using fine slits).



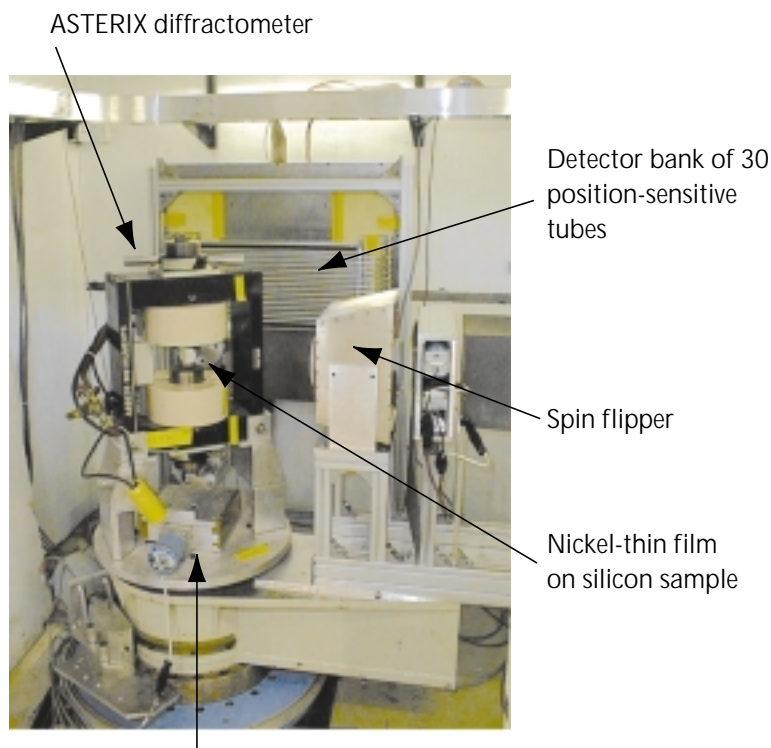
▲ **Fig. 1.** Variation of spectra measured for ASTERIX under different measurement conditions, including (1) tight collimation, (2) without slits in the neutron beam, and (3) with a beryllium filter.

The variation of the red spectrum with wavelength is exactly the same as that measured for SPEAR. Unlike SPEAR, ASTERIX views the moderator through a ^{58}Ni -coated neutron guide. As such, a slower decay in the spectrum is expected when neutrons reflecting from the guide surfaces are permitted to enter the neutron detector (or, more importantly, interact with a sample). The spectrum measured by the neutron detector without any slits in the beam (the beam cross section at the end of the guide is 6 cm by 6 cm) is shown by the blue curve. An upcoming challenge will be to develop experimental techniques to do reflectometry and diffraction measurements that relax requirements for tight collimation. Fig. 2 shows first reflectivity data taken using the new ASTERIX spectrometer. The profile was taken for two sample settings using a single ^3He pencil detector. The variation of the neutron spectrum (shown in Fig. 1) was removed from the measured data to obtain the profile shown in Fig. 2. No background was removed from the measured data.

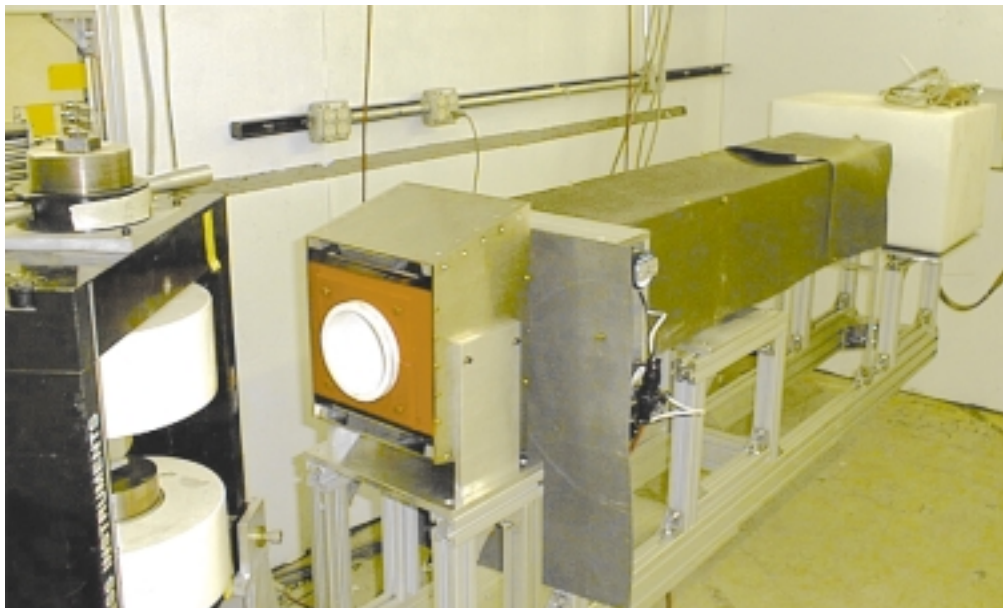


▲ **Fig. 2.** Reflectivity profile of a thin nickel film grown on a 5-cm-diam silicon substrate.

The reflectivity data shown in Fig. 2 were taken from the sample positioned between the pole pieces of the electromagnet (Fig. 3). The diffractometer (courtesy Brookhaven National Laboratory) consists of a World War II 5-in. gun mount from a merchant marine ship, which is now used to position the sample and orient the position-sensitive detector (whose housing is to the right of the picture, which is shown in Fig. 4). Situated behind the electromagnet in



▲ **Fig. 3.** View of the ASTERIX diffractometer on which a large electromagnet rests.



▲ Fig. 4. View of the position-sensitive neutron detector, the polarization analyzer, the slits, and the spin flipper.

Fig. 3 is a second detector—a bank of 30 position-sensitive tubes that are used for measurements of large d-spacing Bragg reflections from single-crystal and polycrystalline materials. To the right of the electromagnet is a radio-frequency gradient-field spin flipper obtained through a collaboration with the Hahn-Meitner Institute. The neutron optical equipment used to filter (for example, the beryllium filter) and polarize (for example, the polarization-analyzer cavity) the neutron beam are out of view to the left of the diffractometer.

In Fig. 4, a pair of slits (to reduce background) are behind the spin flipper, followed by a 1-m-long polarization analyzer (a mirror inside the gray structure) and finally a borated polyethylene shield house for the position-sensitive detector. This detector can be moved from -10° to $+30^\circ$ about the axis of the diffractometer (the vertical axis passing through the electromagnet).

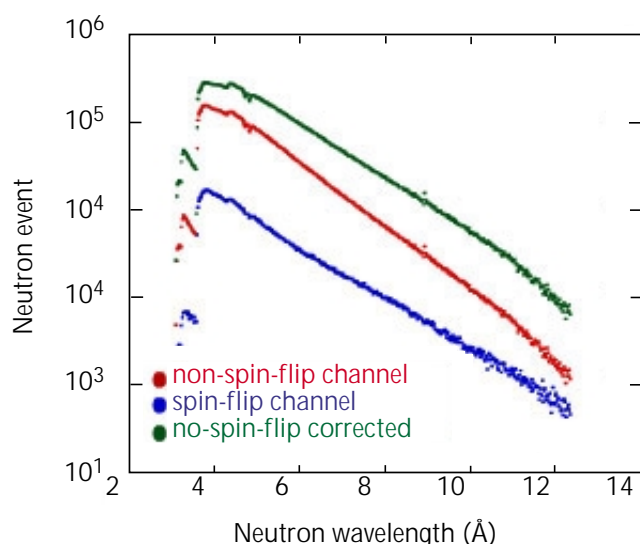
We measured the polarization of the neutron beam by illuminating a second neutron polarizer—the polarization analyzer—with the 2-cm-wide by 12-cm-tall neutron beam from the polarization cavity. Non-spin-flip neutrons (neutrons with spin anti-parallel to the laboratory magnetic field) are transmitted through the relatively thick silicon substrates on which the neutron-polarizing material was deposited. The intensity of the transmitted neutron beam was measured by a ^3He pencil detector. Spin-flip neutrons (those undesirable neutrons with spin parallel to the laboratory magnetic field) were reflected by the polarization analyzer. The intensity

of the reflected neutron beam was measured by a second ^3He pencil detector. The ratio (called the flipping ratio) between the transmitted and reflected neutron-beam intensities (as measured by the two detectors) relates to the polarization of the neutron beam—the larger the ratio, the higher the polarization of the neutron beam.

The raw data obtained across the entire 2-cm-wide by 12-cm-tall neutron beam for non-spin-flip and spin-flip neutrons are shown as the red and blue curves, respectively, in Fig. 5. The transmitted (non-spin-flip) neutrons traveled through the thick silicon substrates of the polarization analyzer, whereas the spin-flip neutrons did not because they were reflected off the surface of the polarizer. As such, we corrected the data for the non-spin-flip channel for losses resulting from absorption (green curve, Fig. 5). We obtained a neutron-beam polarization of 95% by considering the ratio of the green curve (non-spin-flip cross section, corrected) to the blue curve (spin-flip cross section).

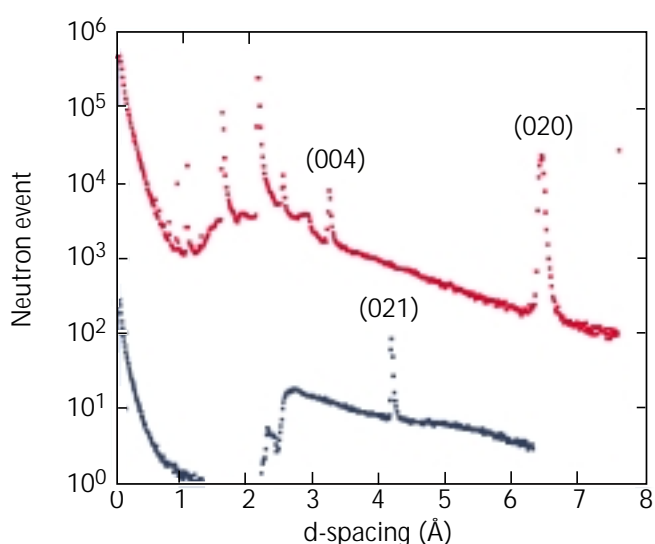
Although we had hoped for better polarization, 95% polarization is typical for neutron spectrometers at pulsed sources and the ADAM spectrometer at the Institute Laue-Langevin. The installation of new polarizing supermirrors from the Hahn-Meitner Institute in the polarization cavity should increase the polarization of the neutron beam to 97%.

During the first week of beam in 2001, we made an important measurement demonstrating the capability of ASTERIX to obtain diffraction patterns from



▲ **Fig. 5.** Polarized neutrons are produced on ASTERIX using novel polarization-cavity technology (novel for pulsed neutron sources!).

small samples in the wide-angle regime in record times. Specifically, we studied a 2-mm x 2-mm x 1-mm LaSrFeO_4 sample. The intensity measured using just one tube in the position-sensitive detector array as a function of d-spacing is shown in Fig. 6 for two sample orientations. In the upper curve, several nuclear Bragg reflections are observed. Nearly 150,000 neutron counts were measured during a 16-h run in the (020) Bragg reflection. We compared the results obtained from ASTERIX with those taken for the same sample on the SCD. For the large d-spacing reflections [those relevant to studies of magnetism and complex (large unit cell) materials], ASTERIX out performed the Single Crystal Diffractometer by more than a factor of 500. The lower curve shows the (021) Bragg reflection that is purely magnetic in origin (due to the antiferromagnetic order in LaSrFeO_4).



▲ **Fig. 6.** Intensity measured as a function of d-spacing for two orientations of a LaSrFeO_4 sample.

This demonstration is important in that we can show that even with the 30-T pulsed magnet operating at a frequency of 1 Hz (thus only one in twenty neutron pulses provide useful information), we still have sufficient neutron flux to observe large d-spacing Bragg reflections. For example, we anticipate that typical experiments using this magnet will require roughly 12 h of beam time (equivalent to 0.4% of the magnet's lifetime). Not only will the high flux of ASTERIX be important to the pulsed-magnet program, the high flux also enables studies of very small samples—samples that might be small due to content of actinides, or due to the availability of neutron isotopes.

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